

## **Chemical and Physical Methods of the Laboratories.**

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## **1. SAMPLE PREPARATION**

### **1.1 METHOD**

Much work has to be done before a sample of any material is ready for analysis. The usual sequence is as follows:

- ◆ weighing and registration of the received sample
- ◆ drying in a laboratory oven
- ◆ splitting of the sample to obtain smaller, representative samples
- ◆ coarse crushing in a laboratory jaw crusher
- ◆ re-splitting into smaller portions
- ◆ fine grinding with a swing grinding mill
- ◆ sieving with different sets of sieves (60  $\mu$ m for analytical purposes)
- ◆ fine re-grinding
- ◆ homogenizing of the final samples with a Turbula-mixer
- ◆ filling into bottles and labeling the samples

### **1.2 APPLICATION:**

All types of material from raw material to cement and concrete

### **1.3 TIME OF PREPARATION**

2- 3 samples per hour can be prepared to 60  $\mu$ m fineness depending on hardness and size of the original sample.

### **1.4 MAINTENANCE AND PERSONNEL**

Installation and service of mills, crushers and mixers can be done by the in-house workshop and electricians. The work has to be done by trained personnel .

### **1.5 SUPPLIERS:**

A. Fritsch & Co., Retsch Laborgeräte, Maschinen fabrik Herzog, Bachofen AG  
Maschinenfabrik for Turbula-mixer

### **1.6 COST OF EQUIPMENT**

Approx. SFr. 140'000 to 150'000 for complete preparation laboratory



## **2. GRAVIMETRY**

### **2.1 METHOD**

Gravimetric analysis or quantitative analysis by weight is the process of isolating and weighing an element or a definite compound of the element in as pure a form as possible. The element or compound is separated from a weighed portion of the substance being examined. A large proportion of the determination in gravimetric analysis is concerned with the transformation of the element or ion to be determined into a pure stable compound which can be readily converted into a form suitable for weighing. The weight of the element or ion may be readily calculated from a knowledge of the formula of the compound and the atomic weights of the constituent elements.

The separation of the element or of the compound containing it may be effected in a number of ways, such as: precipitation methods, volatilisation or evolution methods, electro-analytical methods and miscellaneous physical methods.

One of the most important methods in gravimetry is precipitation, i.e. the constituent being determined is precipitated from solution in a form which is almost insoluble in water so that no appreciable loss occurs when the precipitate is separated by filtration and weighed.

### **2.2 APPLICATION**

Determination of all elements in all kinds of material. The alkalis sodium and potassium are very difficult to determine by gravimetry.

### **2.3 TIME OF ANALYSIS**

Very time consuming! At least five hours for the determination of the four main elements - or 4 samples per day

### **2.4 MAINTENANCE AND PERSONNEL**

Minimum of maintenance required for balances and furnaces. Routine work should be executed by skilled and qualified personnel.

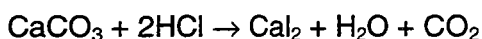
### **2.5 EQUIPMENT:**

No special instruments and apparatus are needed apart from the normal equipment of an analytical laboratory such as glassware, furnaces, balances, etc.

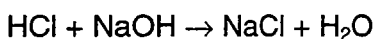
## **3. TITRATION OF CARBONATES**

### **3.1 METHOD**

A sample of a raw material (e.g. limestone) or raw meal is dissolved in a known excess of hydro chloric acid:



The excess of acid is then determined by a so called back titration with sodium hydroxide solution



The end point of the back titration can be found visually by means of an indicator (dye stuff) which changes its colour or by measuring an electrical parameter, i.e. the potential difference between two electrodes dipping into the solution. The result is calculated with a programmable pocket or desk calculator.



### **3.2 APPLICATION**

All types of raw material and raw meal; including all carbonates which are expressed as  $\text{CaCO}_3$  by calculation.

### **3.3 TIME OF ANALYSIS**

10 minutes per sample

### **3.4 MAINTENANCE AND PERSONNEL**

Instruments have to be installed and introduced to the operator by the supplier. Routine work can be performed by trained personnel.

### **3.5 SUPPLIERS**

e.g. Metrohm, Mettler, Radiometer, etc.

### **3.6 COST OF EQUIPMENT**

approx. SFr. 22'000 for semiautomatic titration apparatus.

## **4. COMPLEXOMETRIC TITRATION**

### **4.1 METHOD**

The complexometric determination of many elements is based on a titration with a solution of EDTA (Ethylene Diamine Tetra Acetic Acid) using many special indicators. The end point of the titration is found either visually or with the aid of a spectrophotometer which is more sensitive to colour changes of many of the indicators. The results are calculated from the consumption of the EDTA solution.

### **4.2 APPLICATION**

Ca, Fe, Al, and Mg in cement, clinker, raw meal and raw material such as limestone, clay and sand. Titanium and manganese may be determined also by complexometric titration whereas silica has to be analysed by gravimetry or colorimetry.

### **4.3 TIME OF ANALYSIS**

3 - 4 hours per sample including silica, or 6 - 7 samples per day

### **4.4 MAINTENANCE AND PERSONNEL**

Installation and implementation by the supplier or a chemist. Routine work requires laboratory assistant.

### **4.5 SUPPLIERS**

of spectrophotometer and semiautomatic titrators potentiographs: Metrohm and Mettler

### **4.6 COST OF EQUIPMENT**

ca: SFr. 20'000.



**5. SPECIFIC SURFACE ACCORDING TO BLAINE****5.1 METHOD**

The Blaine air permeability apparatus is used to draw a definite quantity of air through a prepared bed of cement of definite porosity. Number and size of the pores in the prepared bed (cement layer of 2.7 g and 1.5 cm thickness) is a function of the size of the particles and determines the rate of flow through the bed. The cement is filled into a cylindrical cell and pressed by a plunger to form the bed. The cell is attached to the U-tube of a manometer, one arm of which is now slowly evacuated until the liquid reaches the top mark on the glass tube. After opening a valve, the liquid flows back again and passing two other marks, a definite volume of air is pressed through the bed of cement.

The time which is required for the meniscus of the liquid to pass from one mark to the other is measured. It is necessary to use a complicated formula for the calculation of the specific surface, therefore it is advantageous to split this formula into three factors, one of them depending only on the density of the cement, the second one depending on the temperature and the third one being the square root of the stopped time. The first two factors may be taken from a diagram which has to be established once for every Blaine apparatus.

**5.2 APPLICATION**

Cement only! Although the method has been used for the determination of the fineness of various other materials, it should be clear that in general relative rather than absolute values are obtained.

**5.3 TIME OF ANALYSIS**

Double determination for one material takes approx. 20 minutes if the density of the cement is known. Otherwise, additional 45 min. are needed for the determination of the density.

**5.4 MAINTENANCE AND PERSONNEL**

Trained personnel.

**5.5 SUPPLIERS**

e.g. Schiltknecht, Zürich; Tonindustrie for semi automatic instrument.

**5.6 COST OF EQUIPMENT**

SFr. 800 to 6'000 for semiautomatic instrument



**6. DETERMINATION OF SULPHUR AND CARBON DIOXIDE****6.1 METHOD**

The solid sample is mixed with metal powder (copper, iron or tungsten) as an accelerator and burnt in a high frequency or resistance induction furnace at cat 1500°C in a stream of pure oxygen. The gases produced in the chemical reaction during combustion are sulphur dioxide, carbon dioxide and water steam. With different analysers it is possible to determine either SO<sub>2</sub> or CO<sub>2</sub> or even both compounds. The simplest and cheapest method uses a titration with potassium iodate executed in an automatic burette which is connected with the induction furnace. Another possibility is the use of an infrared analyser instead of a burette. The IR analyser determines the sulphur dioxide by its absorption of infrared light and displays the result as percent SO<sub>3</sub>. - Carbon dioxide can be determined with another model which gives the result as total carbon. Instruments for the determination of both SO<sub>3</sub> and carbon by infrared light are also on the market.

The Leco Corporation has another type of instrument for carbon analysis which does not apply infrared absorption but a heat conductivity cell.

**6.2 APPLICATION**

Total sulphur and carbon in all types of solid material, with special instruments or by means of accessories and chemical reagents also in fuels such as coal and oil.

**6.3 TIME OF ANALYSIS**

6 - 12 minutes per sample with sulphur titrator depending on material. 3 - 4 minutes per sample with infrared or heat conductivity analyser

**6.4 MAINTENANCE AND PERSONNEL**

Instruments have to be installed by supplier or specialist. Routine work can be done by trained personnel. Service by the supplier is very important for infrared and heat conductivity analysers.

**6.5 SUPPLIERS**

LECO Corporation (USA) for: Titrator DB 64 and IR 232 for SO<sub>3</sub>, IR 212 and WR 12 for carbon, CS 244 for sulphur and carbon

Leybold-Heraeus (Germany): Series 302 for sulphur and carbon

**6.6 COST OF EQUIPMENT**

Titration ca. SFr. 26'000

Infrared analyser ca. SFr. 50'000 to 70'000

Infrared analyser for C + S ca. SFr. 90'000



## **6.7 ATOMIC ABSORPTION SPECTROMETRY**

### **6.8 METHOD**

The sample has to be dissolved in acid, generally hydrochloric acid. This solution is then sprayed into a very hot flame (ca. 2000.C, for some elements 3000.C) where the solvent evaporates and the chemical compounds dissociate into their atomic components. Most atoms remain in the ground state, a fact which is important for atomic absorption.

The light of a so-called hollow cathode lamp (HCL), the cathode of which consists of the element to be determined is led through the flame with the cloud of mostly neutral atoms. The atoms are able to absorb this HCL-light so that the intensity of the light beam is reduced. The absorption is a measure of the number of atoms in the flame which is proportional to the concentration of the element in the solution or at least the sample.

### **6.9 APPLICATION**

All types of material and all elements of interest in cement chemistry except sulphur, fluorine, chlorine and phosphorus.

### **6.10 TIME OF ANALYSIS**

approx. 4 hours for one analysis including Si, Al, Fe, Ca, Mg, K, Na or 6 analyses per day.

### **6.11 MAINTENANCE AND PERSONNEL**

The method has to be introduced by a specialist or chemist. Routine work can be executed by trained personnel.

Service by supplier should be guaranteed.

### **6.12 SUPPLIERS**

Perkin Elmer, Beckman, Varian, Hilger Watts, Jarell Ash, Instrumentation Laboratory, Pye Uni cam (Philips)

### **6.13 COST OF EQUIPMENT**

SFr. 50'000 to 120'000 depending on spectrometer single or double beam, etc.)

## **7. ATOMIC EMISSION SPECTROMETRY (BY INDUCTIVELY COUPLED PLASMA)**

### **7.1 METHOD**

The sample has to be dissolved in acid very often after a fusion procedure. This solution is then sprayed into a very hot argon plasma, a physical flame of more than 6000.C where the solvent evaporates and the chemical compounds dissociate into their atomic components. The atoms are excited by the high temperature of the plasma and are then able to emit light of certain wave lengths which can be measured and which is proportional to the concentration of the elements to be determined.

### **7.2 APPLICATION**

All types of material and all elements of interest in cement chemistry with the exception of the halogens.



### **7.3 TIME OF ANALYSIS**

approx. 1 hour for determination of 30 to 40 elements and including preparation of the solution.

### **7.4 MAINTENANCE AND PERSONNEL**

The method has to be introduced by a specialist and chemist. Routine work can be executed by very well trained personnel.

Very important it that good and prompt service is guaranteed by the supplier.

### **7.5 SUPPLIERS**

ARL, Jarell-Ash, Instrumentation Laboratory, Jobin-Yvon, Kontron, Philips.

### **7.6 COST OF EQUIPMENT**

SFr. 180'000 to more than 300'000 depending on the instrument: sequential or multi-channel spectrometer.

## **8. X-RAY FLUORESCENCE (XRF)**

### **8.1 ANALYSIS:**

Qualitative and quantitative determination of chemical elements with atomic number higher than 9.

### **8.2 METHOD**

Wave lengths dispersive method or non-dispersive technique (energy dispersive)

### **8.3 PRINCIPLE**

The sample (in form of pressed powder tablet or fused bead) is irradiated by primary X-radiation from an X-ray tube. Atoms and ions of the various elements in the sample are thereby excited and emit their characteristic radiation.

- ◆ Wave length dispersive method

The fluorescence radiation from the excited sample is dispersed on special analysing crystals according to Bragg's law (compare with dispersion of sunlight by a prism into the bands of colour of the visible spectrum). The resolved characteristic radiation of the different elements is analysed individually and measured by appropriate detectors. The intensity of the characteristic radiation is thereby a direct measure of the chemical concentration of the corresponding element.

- ◆ Energy dispersive technique

The fluorescence radiation is measured using a semi-conductor together with an analyser which has an electrical response proportional to the quantum energy of the radiation to be measured.

### **8.4 APPLICATION**

Determination of the chemical composition of raw material, raw meal, clinker and cement. Plant laboratories are mostly equipped with so-called wave length dispersive multi-channel units and make use predominantly of pressed powder tablets; central laboratories often prefer the installation of sequential spectrometers to increase flexibility of analysis.

### **8.5 TIME OF ANALYSIS**

According to the application used: 3 to 20 minutes for 8 elements excluding preparation.



**8.6 LABOUR**

1 well trained man

**8.7 SUPPLIERS**

Philips, Siemens, ARL, Rigaku (Ortec, EDAX)

**8.8 COST OF EQUIPMENT**

SFr. 250'000 - 400 '000

**9. X-RAY DIFFRACTION (XRD)**

**9.1 ANALYSIS**

Qualitative and quantitative determination of crystalline solids

**9.2 METHOD**

A monochromatic X-ray beam is directed upon a fine powdered sample. By varying the angle between sample and incoming beam, the X-rays are reflected from the various crystallographic planes of the minerals present. The reflected X-rays contain the following information:

Angle: identification of mineral

Intensity: concentration of individual mineral

**9.3 APPLICATION**

Raw meal: content of quartz, clays, dolomite, etc.

Clinker: content of free lime, periclase, sulphates, aluminate, ferrite and modifications of  $C_3S$ ,  $C_2S$ ,  $C_3A$ , etc.

**9.4 TIME OF ANALYSIS**

Quantitative 10 minutes (computerised)

Qualitative 1 hour

**9.5 STAFF**

Skilled laboratory assistant, mineralogist, chemist

**9.6 SUPPLIERS:**

Philips (Netherlands), Siemens (W. Germany), Seiffert and Co. (W. Germany), CGR (France), Rigaku (USA, Japan)

**9.7 COST OF EQUIPMENT**

SFr. 80'000 to 200'000

**10. LIGHT MICROSCOPY**

**10.1 ANALYSIS**

Determination of microstructure and mineralogical composition



## **10.2 METHOD**

Samples are examined on:

- ◆ polished sections using reflected light
- ◆ thin sections and loose powders using transmitted light

Qualitative information:

- ◆ phase association
- ◆ texture
- ◆ structure

Quantitative information:

- ◆ phase composition
- ◆ crystallite / grain / granule size

## **10.3 APPLICATION**

Determination of actual clinker composition, e.g. percentage of elite, belite, ferrite, aluminate, free lime, periclase and sulphates

Determination of clinker microstructure to ascertain conditions present during production.

Characterisation of raw mix components (e.g. limestone) Investigation of refractory and concrete damages.

## **10.4 TIME OF PREPARATION**

Preparation: approx. 3 hours:

Interpretation: 1 - 3 hours

## **10.5 STAFF**

Skilled laboratory assistant, mineralogist, chemist

## **10.6 SUPPLIERS**

Wild & Leitz (Switzerland), etc.

## **10.7 COST OF EQUIPMENT**

SFr. 20'000 to 60'000



## **11. THERMAL SHOCK TESTING OF BASIC REFRACTORIES**

### **11.1 METHOD**

Test prisms (35 x 35 x 200 mm) are cut from the supplied brick(s) using a diamond impregnated saw, such as to give a full hot to cold face section. For dolomite products, the cutting should be performed dry; with magnesite bricks wet cutting is possible but requires immediate drying afterwards.

For shock testing, the prepared prisms are heated at 1350°C in a laboratory furnace, for a period of up to 15 minutes, including heating up time. They are then placed vertically (i.e. end-on) upon a water-cooled copper plate, on which they are allowed to cool for 10 minutes. After this time, the now cooled prisms are checked for spalling and crack development. They are then returned to the furnace for more heating up and subsequent cooling cycles until 20 such treatments have been performed, assuming of course that previous destruction has not taken place.

### **11.2 APPLICATION**

Testing of new refractory types and quality control on already delivered ones, in cases where doubt exists as to the quality supplied.

### **11.3 TIME OF ANALYSIS**

For the cutting of the prisms about 1 hour is required with drying taking place overnight. The actual thermal testing requires about 9 hours, the actual number of samples tested depending principally on the capacity of the 1350°C furnace.

### **11.4 MAINTENANCE AND PERSONNEL**

No special requirements with regard to type of equipment and its maintenance. The work can be performed by semi-skilled personnel after a short training period.

### **11.5 COST OF EQUIPMENT**

approx. SFr 10'000 for diamond saw, high temperature furnace and cooling plate.

## **12. THERMAL ANALYSIS - DIFFERENTIAL THERMAL ANALYSIS (DTA)**

### **12.1 METHOD**

Qualitative and semi quantitative determination of caloric changes due to exo- and endothermic reactions in materials subjected to heating; max. temperature: 1500°C.

### **12.2 PRINCIPLE**

Approx. 100 mg of sample are weighed into a Pt-crucible and placed into the DTA furnace. A second crucible with  $\text{Al}_2\text{O}_3$  is used as reference. Both, sample and reference are heated in the same furnace according to a selected temperature gradient. The temperature of sample and reference is measured continuously and the difference is recorded.

To relate temperature difference with energy it is necessary to perform a calibration in the temperature range of interest prior to the measurement.



### **12.3 APPLICATION**

Identification of clay minerals in cement raw materials, investigations on de-carbonisation of carbonates and formation of clinker minerals. Behaviour of circulating elements under different atmospheric conditions.

### **12.4 TIME OF ANALYSIS**

Depending on type of application: 30 minutes to 3 hours.

### **12.5 PERSONNEL**

Skilled laboratory assistant

### **12.6 SUPPLIERS**

Perkin Elmer, Stanton Redcroft, Dupont, Netsch

### **12.7 COST OF EQUIPMENT**

SFr. 70'000 - 130'000

Purchase price depends on whether DTA is combined with DCS and/or TGA.

## **13. THERMAL ANALYSIS - THERMO-GRAVIMETRIC ANALYSIS (TGA)**

### **13.1 METHOD**

Quantitative determination of weight losses or weight gains in materials subjected to heating; max. temperature 1000°C.

### **13.2 PRINCIPLE**

Up to 1g of sample (generally approx. 50 mg) are weighed into a Pt-crucible which hangs on filament of the TGA-balance. The sample is heated in a platinum resistance furnace at a selected heat rate. The weight of the sample is determined and recorded continuously. Weight changes are given in absolute values or in percent of initial weight



**13.3 APPLICATION**

Investigation of decomposition, dehydration and oxidation accompanied by weight changes, e.g. determination of degree of hydration and carbonisation in hydrated clinker or cement (open clinker storage, coating in cement mill or silos); determination of crystal water, e.g. of gypsum, hemihydrate etc.; determination of fillers (components) in blended cements; oxidation of sulphides and organic compounds.

**13.4 TIME OF ANALYSIS**

Depending on type of application: 30 minutes to 3 hours

**13.5 PERSONNEL**

Skilled laboratory assistant

**13.6 SUPPLIERS**

Mettler, Perkin Elmer, Dupont, Stanton Redcroft

**13.7 COST OF EQUIPMENT**

SFr. 60'000 - 80'000 Purchase price may depend on whether TGA is combined with DSC and/or DTA.

**14. THERMAL ANALYSIS - DIFFERENTIAL SCANNING CALORIMETRY (DSC)****14.1 METHOD**

Qualitative and quantitative determination of caloric changes in materials subjected to isothermal or non-isothermal heating; max. temperature approx. 600°C.

**14.2 PRINCIPLE**

Approx. 10 - 100 mg of sample are weighed into a Al or Pt crucible and placed into the sample furnace of the DSC. An empty crucible is used as reference and placed into the reference furnace. Both, sample and reference are subjected to the same temperature program and equal atmospheric conditions. Differences in temperature between sample and reference - due to caloric changes - are compensated by supplying the required amount of electrical energy. The heat of reaction is determined by integration of the energy flow. The kinetics of the reaction may also be determined.

**14.3 APPLICATION**

Transformation of phases: melting, crystallisation, evaporation, volatilisation, changes in modifications. Chemical reactions: decomposition, oxidation, dehydration, hydration, e.g. determination and optimization of gypsum phases in cement, hydration of cement, determination of sulfides in raw materials.

**14.4 TIME OF ANALYSIS**

Depending on type of application: 30 minutes to 1 day.

**14.5 PERSONNEL**

Skilled laboratory assistant



**14.6 SUPPLIERS**

Perkin Elmer, Mettler, Dupont, Stanton, Redcroft

**14.7 COST OF EQUIPMENT**

SFr. 60'000 - 150'000

Purchase price depends on whether DSC is combined with DTA and/or TGA

Ref. PRO MA.86/0015/2



